

The Referee Analyzes - How Good are They?

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Thermo Electron, Portable Elemental Analysis

Workshop on Restricted Substances in Materials,

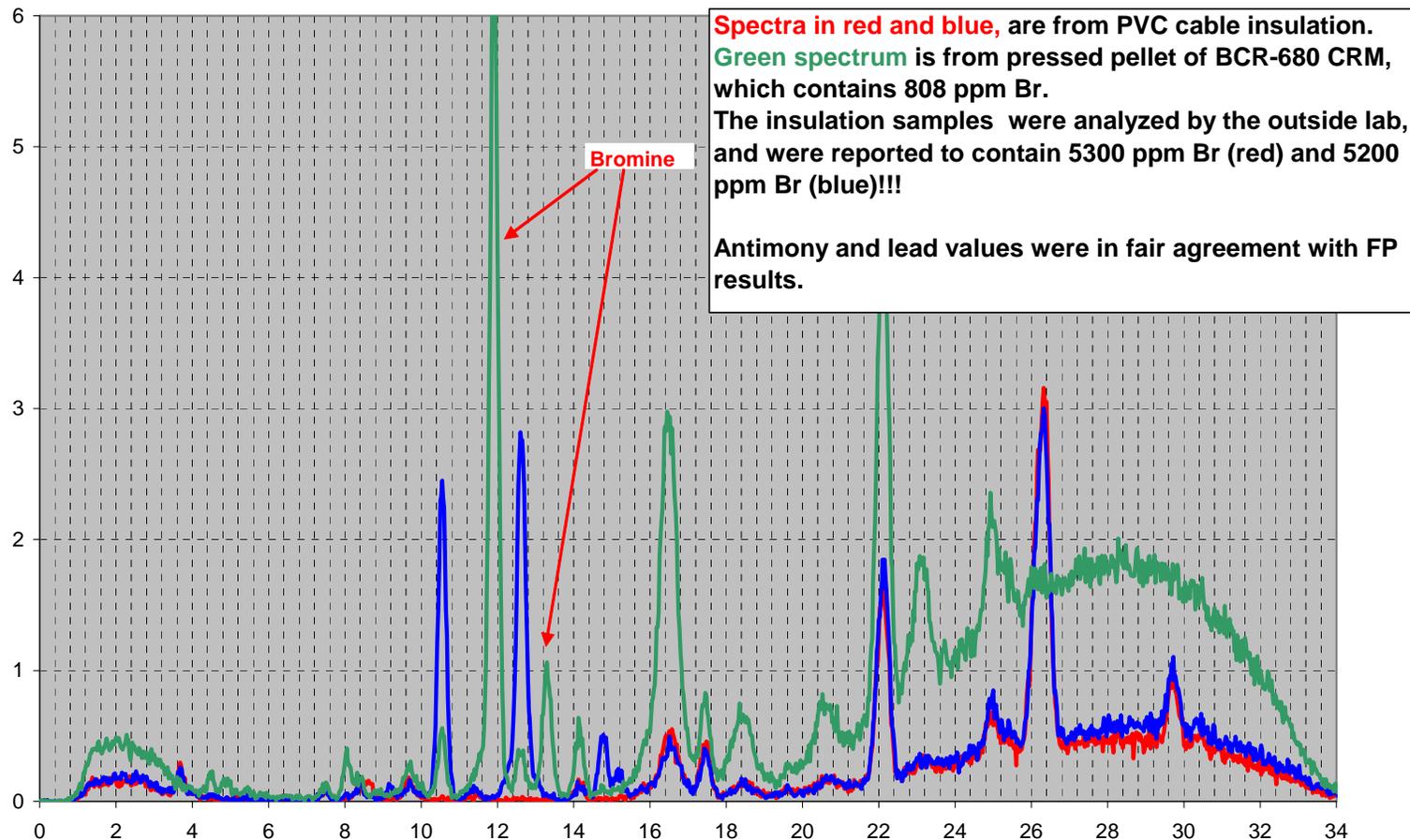
October 5 – 7, 2005, NIST, Gaithersburg MD

The Problem

- *It has been a repeated experience that otherwise reputable analytical service laboratory may very well fail completely when given the task of elemental analysis of polymers*
- *Three examples that follow illustrate the issue*

Example 1 - Where is Bromine?

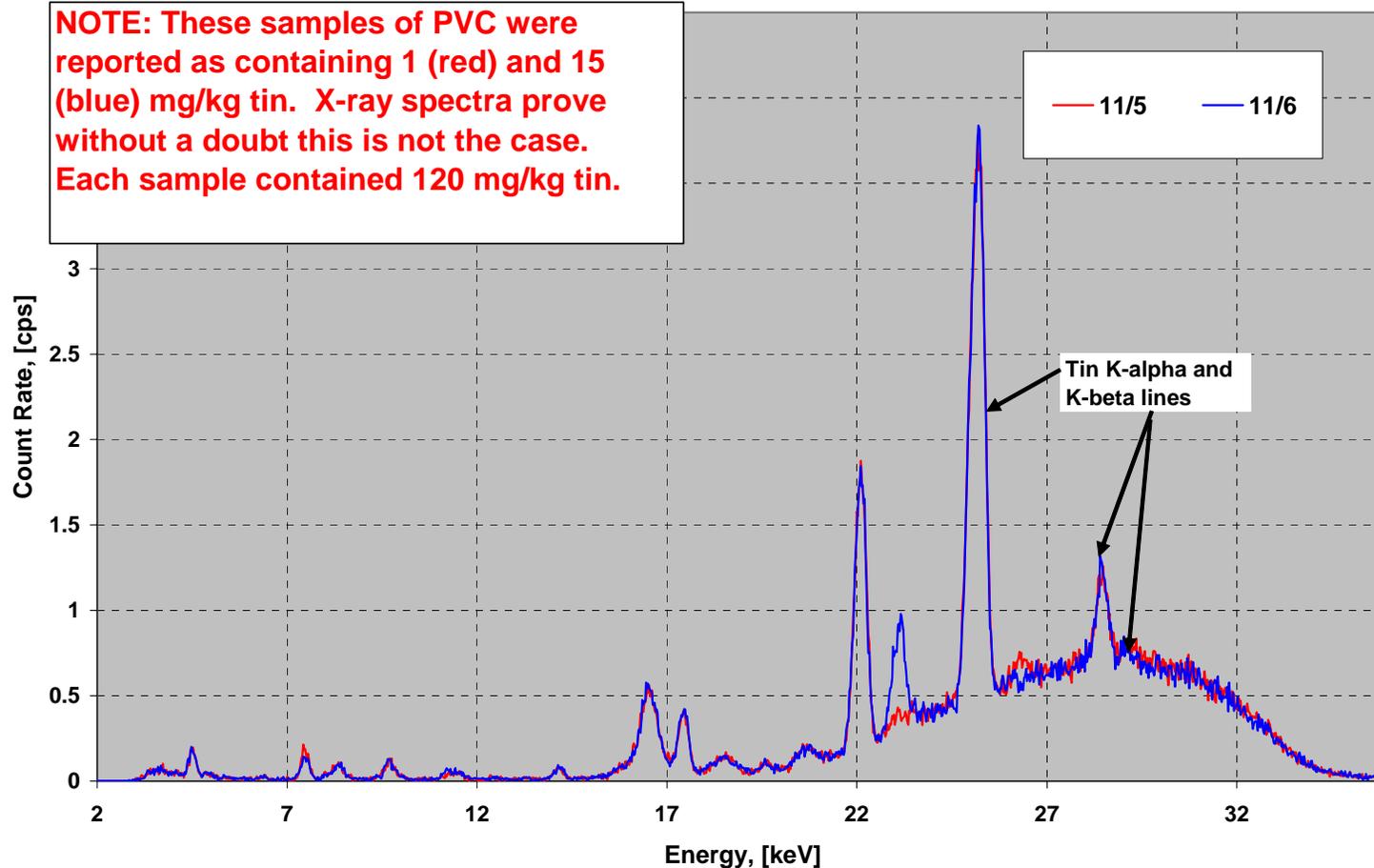
How much can we rely on reference analysis ?
(or Where in the World is Bromine?)



Example 2 - The Lab Misses Tin

X-Ray Spectra of Samples containing Tin

NOTE: These samples of PVC were reported as containing 1 (red) and 15 (blue) mg/kg tin. X-ray spectra prove without a doubt this is not the case. Each sample contained 120 mg/kg tin.



Example 3 - Lab Misses Chromium

Original Data		ICP-OES Analysis			
Serial Number	4-105-3	Sub-sample A of Sample SN 4-105-3		Sub-sample B of Sample SN 4-105-3	
Element	Conc. added, [mg/kg] ^{a)}	Results, [mg/kg] ^{a)}	One Sigma Error, [mg/kg] ^{a)}	Results, [mg/kg] ^{a)}	One Sigma Error, [mg/kg] ^{a)}
Br	2.23	1.90			
Sb	1.99	2.00	0.04	2.01	0.04
Ti	0.51	0.497	0.01	0.497	0.01
Cr	998	837	17	843	17
Pb	800	921	18	965	19
Cd	502	512	10	504	10

After first pass

< 20

After the first pass the lab reported less than 20 mg/kg chromium!!!

Only after they were told what type of compound was Cr in, were they able to repeat the analyzes.

Conclusions

- The labs may have not developed proper analytical procedures to analyze plastics and polymers.
- It appears, the main problem is with selection of proper digestion method.
- If the ICP-OES methods are to be used as ultimate verification “tools” the procedures need to be developed, validated and proliferated so that ICP-OES analyses of polymers may be trustworthy.
- This also shows the need for proper CRMs.